

PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of: Attorney Docket No.: 3081.117US01

Linde et al. Confirmation No.: 9835

Application No.: 10/536,665 Examiner: Wong, Edna

Filed: May 27, 2005 Group Art Unit: 1795

For: PRODUCTION OF STRUCTURED CHROME LAYERS

DECLARATION PURSUANT TO 37 C.F.R. § 1.132

I, Rudolf Linde, declare under penalty of perjury that the following is true and correct to the best of my knowledge, information and belief:

1. I am a named inventor on the above-referenced patent application entitled "Production of Structured Chrome Layers." I have conducted five (5) comparative experiments to the claimed invention, which are summarized in below:

Experiment 1 (according to Example 2 of the present U.S. application no. 10/536,665)

A chromium electrolyte having the following composition was prepared:

Chromium acid anhydride (CrO₃): 250 g/L

Sulphuric acid (H₂SO₄): 2.5 g/L

Ammonium molybdate ((NH₄)₆Mo₇O₂₄ · 4 H₂O): 100 g/L

Methane sulfonic acid: 4 g/L

No fluorides added

A piston ring was introduced into the electrolyte after conventional pre-treatment and electrolytically coated for 30 minutes at 55°C with a cathodic current yield of 11%. A structured hard chromium layer was obtained.

Experiment 2 (according to Example A of Horsthemke et al. / US 6,837,981 B2, without F)

Chromium acid anhydride (CrO ₃):	180 g/L
Sulphuric acid (H ₂ SO ₄):	1.8 g/L (1 % of the chromic acid content)
Molybdic acid (MoO ₃ , comm. grade, 85 %):	90 g/L
Methane sulfonic acid:	2.1 g/L
No fluorides added	

A piston ring was introduced into the electrolyte after conventional pre-treatment and electrolytically coated for 30 minutes at 55°C with a cathodic current yield of 16%. A non-structured hard chromium layer was obtained.

Experiment 3 (according to Example A of Horsthemke et al. / US 6,837,981 B2, with F)

Chromium acid anhydride (CrO ₃):	180 g/L
Sulphuric acid (H ₂ SO ₄):	1.8 g/L (1 % of the chromic acid content)
Molybdic acid (MoO ₃ , comm. grade, 85 %):	90 g/L
Methane sulfonic acid:	2.1 g/L
Fluoride (F ⁻):	0.28 g/L

A piston ring was introduced into the electrolyte after conventional pre-treatment and electrolytically coated for 30 minutes at 55°C with a cathodic current yield of 16%. A non-structured hard chromium layer was obtained.

Experiment 4

A chromium electrolyte having the following composition was prepared:

Chromium acid anhydride (CrO ₃):	250 g/L
Sulphuric acid (H ₂ SO ₄):	2,5 g/L
Ammonium molybdate ((NH ₄) ₆ Mo ₇ O ₂₄ · 4 H ₂ O):	5 g/L
Methane sulfonic acid:	4 g/L
No fluorides added	

A piston ring was introduced into the electrolyte after conventional pre-treatment and electrolytically coated for 30 minutes at 55°C with a cathodic current yield of 23%. A non-structured hard chromium layer was obtained.

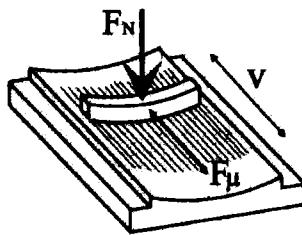
Experiment 5

A chromium electrolyte having the following composition was prepared:

Chromium acid anhydride (CrO ₃):	250 g/L
Sulphuric acid (H ₂ SO ₄):	2.5 g/L
Ammonium molybdate ((NH ₄) ₆ Mo ₇ O ₂₄ · 4 H ₂ O):	100 g/L
Methane sulfonic acid:	4 g/L
Fluoride (F ⁻), added as KF:	0.5 g/L

A piston ring was introduced into the electrolyte after conventional pre-treatment and electrolytically coated for 30 minutes at 55°C with a cathodic current yield of 11 %. A non-structured hard chromium layer was obtained.

2. Afterwards, the relative wear resistance and the seizure resistance of the coated piston rings obtained according to Experiments 1 – 5 were determined by a Rig test (heatable Rig tester of the company Plint). The Rig tester moves a part of a piston ring over a part of a cylinder wall in an oscillating manner. The frequency and the stroke of the movement determine the relative velocity of the piston ring and the cylinder wall:



Conditions:

Measurement of the wear resistance: 23 h, F_N (load) = 450 N, T = 190 °C, f (frequency) = 10 Hz, S (stroke) = 30 mm, ring diameter \varnothing = 78.3 mm.

Measurement of the seizure resistance: F_N (load) = 30 - 800 N, load enhancement = 20 N/5 min, termination at $\mu = 0.3$, T = 120 °C, f (frequency) = 40 Hz, S (stroke) = 4 mm, piston ring diameter \varnothing = 78.3 mm.

In order to compare the wear resistance and the seizure resistance, the coated piston ring according to Experiment 1 was given a value of 100%. A higher percentage indicates a higher wear resistance and the seizure resistance, respectively.

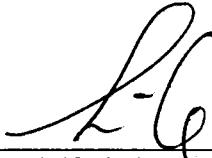
3. The results are shown in the following table:

	Structure of the hard chromium coat	Wear Resistance	Seizure Resistance
Experiment 1	structured	100 %	100 %
Experiment 2	non-structured	50 – 70 %	50 – 70 %
Experiment 3	non-structured	50 – 70 %	50 – 70 %
Experiment 4	non-structured	50 – 70 %	50 – 70 %
Experiment 5	non-structured	50 – 70 %	50 – 70 %

4. The above data shows that cathodic current yields above 12% or the addition of fluoride give hard chromium coatings which do not have the desired structure and, consequently, have a significant lower wear resistance and seizure resistance. Therefore, there is a significant

improvement of the hard chromium coatings prepared according to present claimed invention compared to hard chromium coatings of the prior art.

Executed this 22 day of July, 2009



Rudolf Linde